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[54]	METHOD OF FORMING METALLIC AND CERAMIC THIN FILM STRUCTURES USING METAL HALIDES AND ALKALI METALS		
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[51] [52]	U.S. Cl		

[56] References Cited

U.S. PATENT DOCUMENTS

Field of Search 427/250, 253,

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255.391, 314

3.244.482	4/1966	Culbertson et al
4,812,301		Davidson et al 423/440
4,861,623	8/1989	Ueki et al
5,021,221	6/1991	Gould et al
5,091,209	2/1992	Claverie et al 427/38
5,149,514	9/1992	Sanjorjo et al 423/344
5,175,017	12/1992	Kobayashi et al 427/8
5,453,124	9/1995	Moslehi et al 118/715
5,498,446	3/1996	Axelbaum et al 427/212
5,545,436	8/1996	Saito 427/255.3

5,595,784	1/1997	Kaim et al 427/255.2
5,693,368	12/1997	Ackerman et al 427/253
5,700,519	12/1997	Lam

OTHER PUBLICATIONS

Pierson, "Handbook Of Chemical Vapor Deposition (CVD) Principles, Technology and Applications", Noyes Publications pp. 1–7 and 395–396, 1992.

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[57] ABSTRACT

A new low temperature method for nanostructured metal and ceramic thin film growth by chemical vapor deposition (CVD) involves the use of a low pressure co-flow diffusion flame reactor to react alkali metal vapor and metal halide vapor to deposit metal, alloy and ceramic films. The reaction chemistry is described by the following general equation:

$$(mn)$$
Na+ n MX _{m} \rightarrow (M) _{n} + (nm) NaX

where Na is sodium, or another alkali metal (e.g., K, Rb, Cs), and MX_m is a metal-halide (M is a metal or other element such as Si, B or C; X is a halogen atom, e.g., chlorine, fluorine or the like; and m and n are integers). This reaction chemistry is a viable technique for thin film growth. In one mode, using the precursors of sodium metal vapor, titanium tetrachloride (the limiting reagent), and either argon or nitrogen gases, titanium (Ti), titanium nitride (TiN), titanium dioxide (TiO₂), and titanium silicide (TiSi, Ti₅Si₃, TiSi₂, Ti₅Si₄) thin films have been successfully grown on copper and silicon substrates. Conditions can be adjusted to prevent or minimize gas-phase particle nucleation and growth. Substrate temperatures can also be varied to prevent excessive salt deposition.

18 Claims, 13 Drawing Sheets

